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Fracture mechanisms of a Mo alloyed CoCrFeNi high entropy alloy: In-situ SEM investigation

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ABSTRACT

This paper is focused on an experimental investigation on the fracture property and mechanisms of a Mo alloyed high entropy alloy (HEA) $CoCrFeNiMo_{0.2}$ that demonstrates a promising damage tolerance capacity under room temperature. The employed experimental means is the recently developed in-situ scanning electron microscope (SEM) testing system that allows observation on the deforming material surface in real time and under variable spatial resolutions. A dedicated fracture test on the HEA is performed, and the overall process of crack nucleation and propagation is recorded thoroughly via the in-situ SEM. A remarkable finding of the tested specimen is the elevated steady-state crack-tip-opening angle (CTOA) during the stable crack extension, indicating a satisfactory crack resistance capacity of the studied material. Further analysis on the crack propagation demonstrates that the widespread Cr-rich hard intermetallic particles act as the weak sites for microvoid nucleation, and the high energy ductile fracture mechanism characterized by the microvoid nucleation, growth and coalescence is well identified. Intensified plastic straining along the crack propagation path in a zigzag pattern is remarked, and multiple deformation mechanisms are recognized in the crack tip plastic zone, involving both slip and twinning. The observed microstructural changes are consistent with the macroscopic evaluation on the fracture toughness of the material, and they are accountable for the excellent damage tolerance capacity of the investigated HEA.

1. Introduction

High entropy alloys (HEAs), from a composition-based definition, are normally referred to the alloy systems that are constituted by multi-principal elements in equimolar or near-equimolar ratios. It breaks the traditional "base-element" alloy design restriction, and thus has substantially broadened the realm of alloys [1–3]. A characteristic feature of the HEAs is that it is possessed of a high magnitude of configurational entropy, which normally favors the formation of single-phase solid solutions over competing intermetallic compounds. This characteristic endues the HEAs with some unusual properties and novel mechanisms, as reported by extensive studies [4–15]. The mechanical property is a great concern in the alloy design, e.g., tensile strength and ductility. On this subject, the most commonly studied HEA is CoCrFeMnNi. The investigation [16] shows that the fine-grained CoCrFeMnNi is able to achieve an ultimate tensile strength (σ_{UTS}) of 651 MPa and total elongation (ε_t) of 0.51 at room temperature. The more impressive is its cryogenic tensile properties, with σ_{UTS} = 1099 MPa and ε_t = 0.72 at 77 K. The strength and ductility, two "conflicting" elements in a general sense, are simultaneously and greatly enhanced comparing to that under room temperature condition.

The fracture performance is another great concern for the engineering applications of HEAs, especially when the fatigue load is involved in the service conditions of the material. From a macroscopic viewpoint, the fracture toughness of the material can be evaluated by several optional fracture resistance indicators, for instance, the stress intensity factor *K*, nonlinear energy release rate *J* integral, crack-tip-opening displacement (CTOD), and the crack-tip-opening angle (CTOA). Gludovatz et al. [17] evaluated the fracture toughness of CoCrFeMnNi using both the parameters *K* and *J* integral, revealing the exceptional fracture toughness of the studied HEA. Thanks to the scanning electron microscope (SEM) and electron backscattered diffraction (EBSD) measurements, massive plastic deformations were observed at the vicinity of the crack tip, and the ductile fracture mechanism was demonstrated. This study was pursued by Zhang et al. [18] using transmission electron microscope (TEM) for observing the microstructural evolution details near the crack tip. It revealed that multiple deformation mechanisms were contributing for the outstanding damage tolerance of the HEA CoCrFeMnNi, and in particular the nano-twinning played an important role in the fracture delay

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mechanism. Besides the above works, there are also many other studies reported in recent years that focus on the fracture mechanisms [19,20] and fatigue behaviors [21–23] of the HEAs. Despite the significant body of literature on this topic, the majority of the present investigations were conducted by ex-situ measurements and observations focused on some specific length scales, often very locally via SEM or TEM observation or rather globally by using the macroscopic material parameters (e.g., σ_{UTS}, ε_t, K_{Ic}, J_{Ic}). A trans-scale study connecting the microscale and macroscale is, however, often missed. And some critical in-situ observations on the mesoscale are lacked towards a more comprehensive examination of the local fracture behavior of the HEAs. Hence, the present authors propose to perform an in-situ multiscale SEM investigation on the fracture behavior of the HEA, which is realizable thanks to the development of the in-situ SEM mechanical testing system. This technology is possessed of some unique advantages in the experimental fracture research. First, it is possible to record the complete process of the crack initiation and propagation with accessible global stress-strain state and local deformation manifestation. Furthermore, it enables the observation and analysis of the microstructural evolution and the resulting surface morphology change at variable length scales by the SEM. It is particularly valuable in investigating the local deformation at the crack tip and to explore the underlying fracture mechanisms of the material.

The studied material of the present work is $CoCrFeNiMo_{0.2}$, a HEA prepared via powder metallurgy. The Mo is a minor element for the base HEA composed by the other four principle elements (Co, Cr, Fe, and Ni) in equimolar ratios. The Mo alloyed HEA generally demonstrates a satisfactory combination of tensile strength and ductility, and the relevant deformation mechanisms are studied [24,25]. Its fracture property and mechanisms are, however, rarely reported. The purpose of this study is to investigate the fracture mechanism of the HEA $CoCrFeNiMo_{0.2}$ based on the in-situ multiscale experiment and analysis. The CTOA [26,27] is chosen as the main macroscopic fracture parameter for the measure of the cracking resistance of the material. And the investigation is performed by combining both macroscopic assessment and mesoscopic analysis. The emphasis of the present study is placed on the cracking mechanisms and local deformation characteristics at the crack tip. It is mainly actualized by the in-situ and trans-scale observation during the crack propagation process through the SEM. This paper is organized as follows. First, the studied material and employed methods are presented. Then, the obtained results from the in-situ multiscale experiments are presented in details, and the discussions are provided regarding the cracking behaviors and fracture mechanisms of the investigated material. Finally, some concluding remarks are provided.

2. Material and methods

The studied material is $CoCrFeNiMo_{0.2}$, a HEA constituted by four principle elements Co, Cr, Fe, and Ni in equimolar ratios (23.81 at% for each) with the addition of a minor element Mo (4.76 at%). This configuration provides high mixing entropy, facilitating therefore the production of a single face-centered cubic (FCC) phase. The literature [24,25,28–30] demonstrates that the addition of the element Mo into the CoCrFeNi alloy allows forming hard intermetallic compounds with the other principle elements, and it can bring about precipitation hardening effect on the material. Thus, the Mo is normally considered as a promising element for improving the mechanical property of the CoCrFeNi.

In the present investigation, the $CoCrFeNiMo_{0.2}$ was obtained via the powder metallurgy. The $CoCrFeNiMo_{0.2}$ pre-alloyed powders were prepared through the gas atomization method. Then, the as-prepared powder was filled into a stainless steel can, which was degassed at 500 °C for 12 h and sealed in vacuum. After that, the encapsulated powders were pre-heated at 1473 K for 1 h and immediately subjected to hot extrusion with an extrusion ratio of 9.5 and a velocity of ~ 10 mm/s under the 2500 T hydraulic press. Finally, the billets were cooled in air.

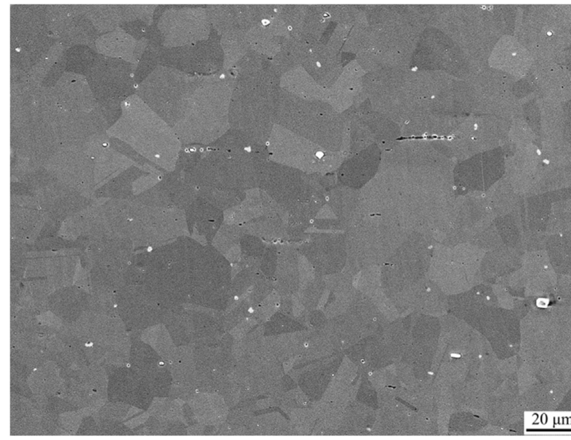

Fig. 1. The SEM image of the as-received HEA $CoCrFeNiMo_{0.2}$.

The surface of the as-received $CoCrFeNiMo_{0.2}$ specimen was treated by mechanical polishing followed by electrolytic polishing for the microstructure observation. The SEM micrograph of the polished specimen surface is illustrated in Fig. 1. It shows that the formed FCC phase has a medium grain size about 10–20 μm. It can be also noted that a certain amount of the particles (in white color) are randomly distributed in the matrix. The diameter of the coarse particles can achieve 3 μm and above, accompanied by fine particles with the diameter around 1 μm. The energy dispersive spectrometer (EDS) analysis was conducted on the observed particles, and demonstrated that the observed particles precipitated in the FCC matrix were mainly the Cr-rich intermetallic compounds, as shown in Fig. 2.

The mechanical property of the $CoCrFeNiMo_{0.2}$ was measured through the tensile tests at room temperature. The tensile testing was conducted under the displacement-control mode with a constant strain rate of 1×10^{-4} s^{-1} by using the MTS 809 hydraulic mechanical testing system. Fig. 3 shows the obtained engineering stress-strain curve of the tested specimen. A typical elasto-plastic deformation behavior is remarked. The yielding occurs at about 400 MPa without apparent yielding plateau. And then it is followed by a long work hardening stage. The elongation to failure can attain 55.6%, with the ultimate tensile strength about 781 MPa. This combination of strength and ductility is considered rather satisfactory in the HEAs.

The present paper is focused on the exploration of the fracture mechanisms of the HEA $CoCrFeNiMo_{0.2}$ based on the in-situ multiscale experiment. Thus, a dedicated experimental protocol was designed to

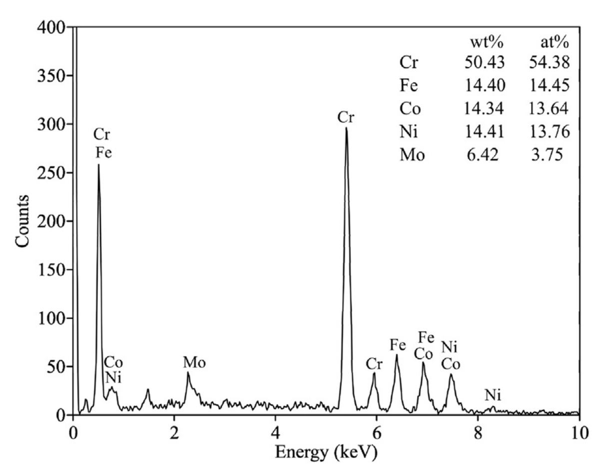

Fig. 2. The EDS analysis on the observed particles of the as-received specimen showing that they are mostly the Cr-rich intermetallic compounds.